

ama  
6-12-92

ADD 406558

E. I. du Pont de Nemours & Co., (Inc.)  
Technical Services Laboratory      Polychemicals Department

TR No. 84

March 1961

TECHNIQUES AND APPLICATIONS FOR BONDING  
AND SEALING TEFLON® 100 FEP RESIN

ABSTRACT

This report summarizes various techniques for bonding and sealing Teflon® 100 FEP resin to itself and to other materials. Techniques included are: 1) bonding to flat surfaces, 2) encapsulation and sealing to metal shapes, and 3) special techniques such as spin welding, film wrapping, hot gas welding, and heat shrinking. Typical applications where a bond or seal is important are discussed.

Work by: E. A. Stecca and G. H. Bowers (R & D)

Report by: (E. A. Stecca)

W. R. No. 244-2883

Distribution

R. B. Akin - TSL  
R. E. Brooks - TSL  
E. D. Champney (2) - D13068  
G. F. Curtin - ESL  
I. du Pont, Jr. - TSL  
H. J. Haon - D12066  
W. B. Happoldt (3) - D13094  
R. E. Maier - ESL

W. B. Thompson - TSL  
W. B. Watkins - D13064  
Marketing Services - D13133  
L. T. Bunn - D13068  
D. E. Eells (10) - Chicago  
R. B. Fehr - Phila. (10)  
G. L. Moore (8) - L. A.  
G. C. Nielsen (12) - N. Y.

19960424 065

DISTRIBUTION STATEMENT A

Approved for public release;  
Distribution Unlimited

® Du Pont Registered Trademark

DEPARTMENT OF DEFENSE  
PLASTICS TECHNICAL EVALUATION CENTER  
PICATINNY ARSENAL, DOVER, N. J.

UNCLASSIFIED//FOR RELEASE

DTIC QUALITY INSPECTED 1

2073

@str@

(ALL)

?04PL-002073

-- TECHNICAL REPORTS STATISTICS PAGE 1 OF 2 JUN 12, 1992

--TOTAL-SEARCH FINDS**-----	1	ARMY--	0
-- FIRST LEVEL FINDS**-----	1	NAVY--	0
-- FIRST AND SECOND LEVEL FINDS**----	0	AF----	0
-- 1+2+3 LEVEL FINDS**-----	0	OTHER--	0

@dsr@

1f

end

w

-- 1 OF 1

-- \*\*\*DTIC DOES NOT HAVE THIS ITEM\*\*\*

-- 1 - AD NUMBER: D406558  
-- 3 - ENTRY CLASSIFICATION: UNCLASSIFIED  
-- 5 - CORPORATE AUTHOR: DU PONT DE NEMOURS (E I) AND CO WILMINGTON DEL  
POLYCHEMICALS DEPT  
-- 6 - UNCLASSIFIED TITLE: TECHNIQUES AND APPLICATIONS FOR BONDING AND  
SEALING TEFLON 100 FEP RESIN.  
-- 8 - TITLE CLASSIFICATION: UNCLASSIFIED  
--10 - PERSONAL AUTHORS: STECCA,E. A. ;  
--11 - REPORT DATE: MAR , 1961  
--12 - PAGINATION: XXXXXX MEDIA COST: \$ 5.00  
--14 - REPORT NUMBER: TR-84  
--20 - REPORT CLASSIFICATION: UNCLASSIFIED  
--27 - ABSTRACT: THIS REPORT SUMMARIZES VARIOUS TECHNIQUES FOR BONDING  
AND SEALING TEFLON 100 FEP RESIN TO ITSELF AND TO OTHER MATERIALS.  
TECHNIQUES INCLUDED ARE: 1)BONDING TO FLAT SURFACES, 2)  
ENCAPSULATION AND SEALING TO METAL SHAPES, AND 3)SPECIAL TECHNIQUES  
SUCH AS SPIN WELDING, FILM WRAPPING, HOT GAS WELDING, AND HEAT  
SHRINKING. TYPICAL APPLICATIONS WHERE A BOND OR SEAL IS IMPORTANT  
ARE DISCUSSED. (AUTHOR).  
--28 - ABSTRACT CLASSIFICATION: UNCLASSIFIED  
--30 - ANNOTATION: TECHNIQUES AND APPLICATIONS FOR BONDING AND SEALING

-- TEFLON 100 FEP RESIN.

--33 - LIMITATION CODES: 1

--35 - SOURCE CODE: 402918

--36 - ITEM LOCATION: PLASTC

--41 - TYPE CODE: 4

--42 - IAC ACCESSION NUMBER: PL-002073

--43 - IAC DOCUMENT TYPE:

-- PLASTEC-HARD COPY

--44 - IAC SUBJECT TERMS: P--(U)TEFLON, BONDING, PTFE, ENCAPSULATION,  
FEP, FILMS, HOT GAS WELDING, SEALING, PEEL STRENGTH, SPIN WELDING,  
ZZ UNLIMITED.;

---\*\*\*\*\*

-- <<ENTER NEXT COMMAND>>

## TABLE OF CONTENTS

	<u>Page</u>
Introduction. . . . .	1
Summary . . . . .	1
Definitions . . . . .	2
I. Bonding to Flat Surfaces. . . . .	4
A. Compression Bonding . . . . .	4
1. FEP Laminates . . . . .	4
Applications. . . . .	6
2. FEP Interlayer Constructions. . . . .	6
Applications. . . . .	6
B. Adhesive Bonding. . . . .	7
II. Encapsulating and Sealing to Metal Shapes . . . . .	8
A. Sealing to Inserts (FEP encapsulates metal insert) . . . . .	8
1. Press Fitting . . . . .	8
2. Induction Heating . . . . .	8
Applications. . . . .	9
3. Molding . . . . .	10
Applications . . . . .	13
B. Sealing to Outer Metal Shell (FEP encapsulated by exterior shell) . . . . .	13
1. Press Fitting . . . . .	13
2. Molding . . . . .	14
Applications. . . . .	16
III. Special Techniques . . . . .	17
A. Spin Welding . . . . .	17
B. Film Wrap Process . . . . .	19
C. Hot Gas Welding . . . . .	20
D. Heat Shrinking. . . . .	21
Applications. . . . .	21

## INTRODUCTION

Complementing the family of Teflon® TFE-fluorocarbon resins is "Teflon" 100 FEP resin, a copolymer of tetrafluoroethylene and hexafluoropropylene. The molecular structure of FEP resin retains essentially all of the properties of TFE resins while providing a melt viscosity low enough for processing in conventional thermoplastic equipment. In addition, this melt flow characteristic permits heat bonding and sealing to itself and to other materials.

Development work was needed to define suitable techniques and optimum conditions for bonding shapes of FEP resin to meet various seal requirements. This report summarizes the various techniques developed for bonding and sealing FEP resin and reviews typical applications where an important bond or seal problem was solved.

## SUMMARY

Heat and pressure are the primary requirements for obtaining a melt bond or fusion to a substrate with "Teflon" 100. Various design considerations and techniques which utilize the principle of melt bonding to obtain good seals with FEP resin are considered. "Water tight" and "moisture proof" seals are readily achieved. Further refinements of this technique may produce a "hermetic seal".

Induction heating metals in contact with FEP resin is a useful technique for generating a melt film and bond. Injection molding to encapsulate inserts is very effective. Metal surface pretreatments help improve bond strengths. Designs which take advantage of the shrinkage characteristics of FEP resin are effective in generating pressure against the surface to be bonded; this approach has led to several methods of encapsulation. Special techniques were developed which rely on melt bonding, including spin welding, film wrapping, and hot gas welding.

There may be applications for a seal wherein the high temperature of FEP required for a melt bond cannot be tolerated. In these instances the use of adhesives with a prepared surface of FEP is effective. Methods of achieving a mechanical seal by press fitting are also useful.

## DEFINITIONS

In this report a bond is defined as fusion or adhesion to a substrate. Bond or peel strengths will vary depending upon the extent of fusion at the points of contact. A seal is defined as that construction which prevents passage of a gas or liquid. The quality of a seal is generally determined by the leakage rate of a specific fluid for a given pressure differential. A bond without a perfect seal may be adequate for some applications. At the same time, a seal may or may not require a bond. For example, a simple press fit could give a water-tight seal. Environmental conditions, electrical strength, and thermal cycling, will influence the choice of appropriate technique for achieving an acceptable seal.

The term "hermetic" is frequently used to define an "air-tight" seal in which the leakage rate is extremely small. The viscosity of the fluid used for testing and the pressure differential employed are two variables which will affect the measured leakage rate. Use of a mass spectrometer is probably the only accurate method of testing a true hermetic seal. In this report, we will consider a hermetic seal as having an air leakage rate less than 0.003 cc/hr. at 40 psi pressure differential. This rate represents a typical requirement for hermetic seals in electronic equipment.

There are applications where a "moisture proof" seal is required. Seals in electrical equipment, in particular, must be moisture proof to prevent electrical shorting across conductors. Moisture here is defined as that water condensed from air. A hermetic seal is moisture proof. The allowable leakage requirements for a "moisture proof" seal, however, are probably somewhat greater than for a hermetic seal. The maximum allowable moisture penetration through a seal will depend on the requirement of the application and the relative humidity of the environment.

Another seal frequently referred to is a "water-tight" seal. This is defined as that construction which prevents passage of water when the unit is partially or completely submerged in water. "Water-tight" is intended to include all liquid systems. A "water-tight" seal is the simplest to achieve at normal operating pressures. Merely from a difference in viscosities, 1 centipoise (cps.) for water versus 0.018 cps. for air, we can see that water would have a volume leakage rate considerably less than air across a seal under the same pressure differential. Also, the test for water leakage is usually conducted at a lower pressure (probably atmospheric) than for air leakage tests. This smaller driving force will lower the leakage rate for water even further.

Finally, when "Teflon" is used in a seal, there is the advantage of having a nonwetable surface where little capillary action will occur. No specific tests of seals submerged in water were made in this study since from air leakage rates obtained, one could estimate a water leakage rate.

## I. BONDING TO FLAT SURFACES

### A. Compression Heat Bonding

#### 1. FEP Laminates

Bonding FEP resin to metal substrates is readily performed using heat and pressure without an adhesive. Although very satisfactory melt bonds have been achieved with a number of metal surfaces, it is readily apparent that no simple formula of bonding conditions will apply to all applications considered. Processors may need to alter conditions for each successful application.

Preparation of laminates of FEP resin is generally limited to batch-wise compression lamination of film or sheeting to various substrates or to direct compression molding of resin onto the substrate. Film in thicknesses ranging from 1/2 mil to 40 mils is available commercially from the Film Department of Du Pont.

Conditions required to obtain satisfactory bonds include (1) a temperature above the resin melting point (545° F.), (2) pressures in the range of 25-1000 psi, (3) a dwell time of 1-2 minutes, and (4) selective surface pretreatment of the metal substrate.

It is important that a uniform pressure be maintained across the press platens. Bond strengths improve with increased temperature to about 600° F., which appears optimum. Above this temperature, heat striations or bubbles, probably caused by release of volatiles in the resin, may develop and degradation can occur. A slip sheet is helpful in preventing sticking of molten FEP resin to the press platens. Aluminum foil, "Teflon" TFE impregnated glass cloth and silicone rubber have been used successfully as slip sheets below 620° F. Above this temperature a thin film of silicone release compound or talc should be applied to these materials to insure adequate release of the slip sheet. The glass cloth will leave a slightly matted surface on the FEP film.

Pressures applied will primarily depend upon the characteristics of the substrate. Bonding to aluminum, for instance, requires higher pressures than for steel. Bond pressures should not exceed the point where resin extrusion or deformation occurs. While pressures higher than the minimum required to obtain fusion do not significantly improve bond strength, they may permit bonding at a slightly lower temperature because of better heat transfer. Laminates should preferably be cooled under pressure below the melting point of FEP resin (to about 300° - 400° F.). Water quenching is equally effective if faster cycles are desired.

Satisfactory bonds have been achieved with substrates such as steel, copper, aluminum, glass, and "Teflon" TFE resin. Metal surface pretreatment to improve wetting action of the FEP will improve bond peel strengths. Oxidation of the surface by air or by chemicals to produce an oxide coating is sometimes effective. Sandblasting frequently improves bond strengths as well. The surface should be cleaned with a solvent to remove all traces of dirt, grease or other contaminations. To improve bond strengths even further, particularly if the laminate will be exposed to severe thermal cycling, the substrate can be coated with a "primer" of TFE or FEP resins. "Primers" consist of dispersions of "Teflon" or formulations based on dispersions which are spray coated, dried, and then fused or sintered to the substrate.

Table I outlines the level of bond strengths achieved with FEP resin and various substrates.

TABLE I  
LAMINATES  
Peel Strength, #/in.  
(10 mil FEP film)

<u>Substrate</u>	<u>Type of Treatment</u>	<u>Peel Strength, #/in.</u>
Steel	None	1-3
	Sandblasted & degreased	3-8
	Sandblasted, degreased, and oxidized	5-10
	TFE primed	5-10
	FEP primed	6-12
Copper	None	< 1
	Chemically oxidized	3-10
Aluminum	None	< 1
	Chromic acid	5-6
	FEP & TFE primed	10-15

Laminates of 20 mil FEP resin were made with steel, aluminum, and copper by compression molding FEP to the substrate at 650° F. and 250 psi followed by water quenching. The laminates were exposed to thermal cycling conditions consisting of 10 minutes in dry ice, 10 minutes at room temperature, 10 minutes at 450° F. and return to room temperature. Three surface pretreatments were evaluated, including (1) sandblasted degreased surface, (2) sintered coating of a TFE primer finish\*, and

\* The primers evaluated were 850-201 for steel and 850-202 for aluminum, sold by the Fabrics & Finishes Department of Du Pont.



(3) fused coating of FEP resin primer made from an experimental mixture of FEP dispersion and potassium chromate.

FEP primed substrates had the best bond peel strengths after 10 thermal cycles. Steel was measured at 7-9 #/in., aluminum at 10-13 #/in., and copper at 4-9 #/in.

#### a. Applications

Printed circuits are an ideal application for laminates of FEP resin and metal substrates since the excellent electrical properties of FEP are utilized. The technique is also applicable in preparing protective coatings and in lining of equipment. Here the chemical resistance of FEP resin is an important property.

### 2. FEP Interlayer Constructions

Where the high temperature characteristics of TFE are required in a laminate structure, FEP resin can be used as a heat bonding agent. Useful product forms consist of laminations where the FEP resin is used essentially as a bonding material either between two layers of TFE resin or between TFE resin and a metal substrate, glass fabric, or asbestos cloth. Heat seals with bonds as strong as the film itself are possible.

#### a. Applications

Sheets of TFE resin could be bonded together by overlapping the edges and using an interlayer of FEP film. One-eighth inch sheeting of TFE resin was readily heat bonded in laboratory tests using 10 mil FEP film and a 1-1/2 inch overlap. The strength of the bond was equal to the FEP yield strength.

Patching of wire insulated with TFE resin is another application successfully performed using an FEP film interlayer construction. After scarfing the patch area and exposing bare wire all around, a wrap with 2 mil FEP film was made. The film was wrapped over the TFE insulation at either end of the patch area. Wrapping with unsintered TFE tape over the FEP film completed the patch. The patch was then fused by pressing in a 720° F. mold for about five seconds. Without the FEP film, there was poor adhesion of the TFE tape to the insulation, and electrical failures resulted at the scarf joint. With the FEP film, patches were made which exhibited good mechanical strength and were not affected by heat aging at 480° F. for 4 hours. Successful patches passed a dielectric strength test of 2.5 KV for five minutes both before and after a mechanical bend test, a water soak, and the heat aging process.

## B. Adhesive Bonding

The antistick properties of "Teflon" prevent conventional cementing techniques. Therefore, in order to use an adhesive to bond FEP resin to itself or other materials, the surface of the FEP must first be treated or coated to make it receptive to the adhesive. The same technology developed for TFE resin is applicable to FEP resin.

Several chemical resin-etching solutions,\* usually containing metallic sodium, have been devised which presumably extract fluorine atoms from the surface of the "Teflon", thus leaving a carbonaceous film which will accept an adhesive. The treatment can be applied merely by dip coating. This treatment on FEP resin will give bond strengths equal to chemically treated TFE resin. Patents covering this method of treatment have been issued to Minnesota Mining and Manufacturing Company (U. S. Patent 2,789,063), to General Motors Corporation (U. S. Patent 2,809,130) and perhaps others.

Another method of preparing a bondable surface is by graft copolymerization. The process consists of polymerizing monomer A onto polymer B ("Teflon") in such a way that polymer A constitutes a "branch" or "trunk" of polymer B. The plastic selected for grafting to the "Teflon" is one which will accept most conventional adhesives. While this method has primarily been demonstrated as a continuous process for wire insulation, it is adaptable to other shapes and configurations, such as tape and sheeting. (Radiation Applications Incorporated, Long Island, New York, has marketed wire insulated with TFE resin which has been treated by graft polymerization.) To our knowledge this technique of preparing a bondable surface has not been used extensively with FEP resin.

Another pretreatment relies on a mechanically treated "sand paper surface" to which adhesives can lock. The sample of "Teflon" is cleaned and dipped into colloidal silica, then dried and sintered. Heat implants the surface of the "Teflon" resin with the fine silica particles which act as anchors to adhesives. (Colloidal silica dispersions are available from the Fabrics and Finishes Department of Du Pont.)

Once the surface of FEP resin has been properly treated, any number of adhesives can be used. The choice will depend on the characteristics of the adhesive itself coupled with the requirements of the application desired.

Another approach is to use cementable film of FEP resin which is commercially available from the Film Department of Du Pont. No further treatment is necessary to bond this film with commercial adhesives.

---

\* Surface etching preparations are available from Acton Laboratories, Newark, N. J.; Chemineers, Inc., Los Angeles, Calif.; W. L. Gore Associates, Newark, Del.; and Joclin Manufacturing Co., Wallingford, Conn.

## II. ENCAPSULATING AND SEALING TO METAL SHAPES

### A. Sealing to Inserts

#### 1. Press Fitting

One of the simplest methods of obtaining a seal between a metal insert and a shape of FEP resin is by press fitting. A minimum of a 5 to 10 mil interference will result in a tight fit without distortion or scaling of resin. Greater interference will impose stresses that eventually will relax due to cold flow of the resin. At best, this technique will produce a water-tight seal. Where a seal with lower leakage is required, variations of this technique can be used. Extreme thermal cycling will, for instance affect a simple press fit since expansion of FEP resin will relieve the stress imposed by press fitting and a loss of seal can result.

#### 2. Induction Heating

The seal obtained by press fitting will be greatly improved if the metal insert is induction heated sufficiently to melt the resin immediately surrounding it. Induction heating consists of applying a rapidly changing magnetic field around the insert which causes an extremely fast build-up of heat in the metal. FEP resin, being a nonpolar dielectric material, is not affected. Sufficient heat is then transferred from the metal to the resin by conduction in a few seconds time to obtain a tight seal.

The power input of the induction heater and dwell time must be controlled so as to just melt the resin surrounding the insert, without distorting the resin or causing excessive melt flow. A current of 150 milliamps for 5 to 8 seconds, for instance, was adequate for generating a melt film on a 1/8 inch diameter steel pin inside a 1/2 inch diameter rod of FEP resin.

The type of metal used as an insert, its mass, and the distance from the induction heating coil are variables affecting the rate of heat build-up. Magnetic or ferrous materials will induction heat most rapidly. Nonmagnetic metals, such as aluminum, copper, tantalum and nickel, have been induction heated successfully, but a higher power input and longer dwell time are required.

The amount of adhesion and bond strength resulting from this technique will be determined by the degree to which the FEP resin wets the metal surface. Several metal surface pretreatments were evaluated including sandblasting, degreasing,

oxidizing, and coating with a TFE primer. The best seal, and one which would withstand thermal cycling, was obtained with pins that were sandblasted and coated with a TFE primer.

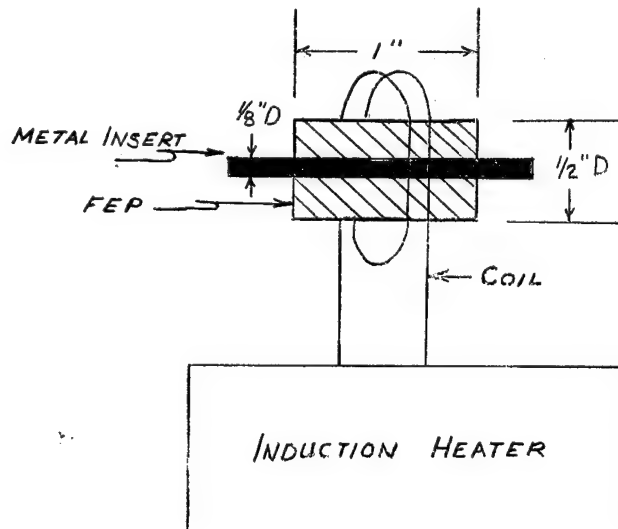
(Fabrics & Finishes Primer 850-201 was used successfully on steel pins.) The primer coat was dried and sintered on the pin before press fitting.

Since limited pressure is available for bonding by this technique, care must be exercised in avoiding excessive melt flow of resin at the ends of the construction, otherwise the stress imposed by the original press fit is quickly relieved. Furthermore, to get good adhesion and fusion to the TFE primer coat, temperatures above the gel state of TFE (above 621° F.) should be reached. This requires a longer dwell time than necessary to merely achieve a melt of FEP resin. A primer coating made from FEP dispersion or by fluid bed coating would eliminate this problem.

#### a. Prototype Applications

Two prototype applications were evaluated using this technique. Both utilized the FEP resin as an insulating material around an insert, one for use in an electrical connector, the other in a capacitor. The configuration of the connector application is sketched below.

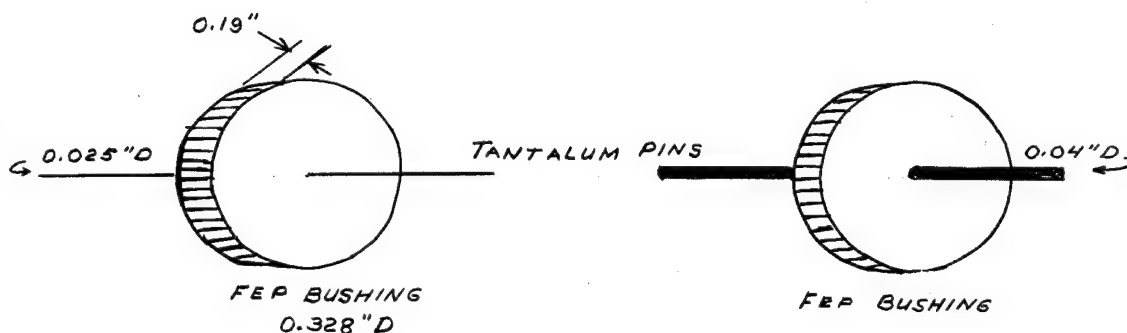
Figure 1  
Prototype Connector



With a TFE primer coating, a seal and definite evidence of bonding occurred after induction heating, but results were erratic and not completely reproducible. When a bond occurred, however, air leakage rates less than 0.2 cc/hr. were recorded. The bond withstood thermal cycling conditions (one hour at dry ice temperature ( $-65^{\circ}$  F.), two hours at room temperature, one hour at  $400^{\circ}$  F. and return to room temperature).

In the capacitor application, the configuration was similar but the metal insert was considerably smaller in diameter. The FEP bushings too were shorter in length. Two sizes of lead wires made of tantalum were evaluated. The configurations are shown below.

Figure 2  
Prototype Capacitor Bushing



Both wires were press fitted into holes machined in the bushing to give a 5-mil interference. Both a TFE primed and unprimed tantalum surface were evaluated.

The test of the seal for leakage consisted of measuring the weight loss of a glycol electrolyte passing through the seal during 1000 hours exposure at  $185^{\circ}$  F. and at  $257^{\circ}$  F. until failure. Leakage less than 10 milligrams per 250 hours exposure was considered satisfactory. Some samples passed this series of leakage tests, although, because of limited area of surface contact in the seal, they were quite delicate and required careful handling to avoid twisting the lead wire free from the bushing.

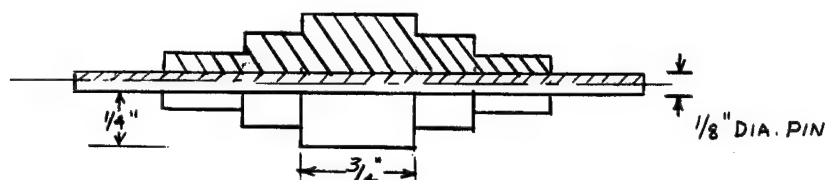
### 3. Molding

The technique which will give the best and most consistent seal of FEP to a metal insert is to injection mold resin so as to encapsulate the insert directly. Here the seal is aided by the high pressures of the molding operation and subsequent melt shrinkage of the resin. Treatment of the metal surface

and preheating before molding are successful techniques employed to achieve a wetting and fusion of melt with the metal insert. Post molding induction heating is also helpful.

Various metal surface pretreatments were employed on 1/8 inch diameter steel, aluminum, and copper pins. They included degreasing, sandblasting, knurling, priming with TFE and FEP resins and combinations of these. The pins were encapsulated with resin in a step cylinder mold. Dimensions and configuration are shown below.

Figure 3  
Step Cylinder Encapsulation



Samples were tested by placing the cylinders in a pressure test apparatus; one side was pressurized and the other was evacuated to give a net pressure drop of 40 psi. Air leakage between the FEP resin and the encapsulated pin was seen as an increase in pressure on the vacuum side. A mercury manometer was employed, and readings were corrected to room temperature and atmospheric pressure.

Samples were tested "as molded", after three thermal cycles and in most cases after an induction heating process. Thermal cycles consisted of one hour at dry ice temperature ( $-65^{\circ}\text{F.}$ ), two hours at room temperature, one hour at  $400^{\circ}\text{F.}$  and return to room temperature. Testing lasted a minimum of 24 hours and results were recorded as a rate in cubic centimeters per hour (cc/hr.).

Reproducible seals with leakage rates in the range of 0.01 to 0.1 cc/hr. were achieved with steel, aluminum and copper inserts. A minimum metal surface preparation consisting of sandblasting and degreasing is necessary if post-molding induction heating is not performed. The use of a "Teflon" primer did not appear to significantly improve the quality of this seal. The metal should be heated before molding (at least  $400^{\circ}\text{F.}$  and preferably closer to melt point of FEP), to achieve maximum fusion of the resin to the insert. From the data obtained it appears that a moisture-proof seal is readily achieved and that further refinements may produce a hermetic seal.

Thermal cycling and testing at increased pressures will result in increased leakage rates across the seal. This is clearly illustrated by the following:

TABLE II  
MOLDED INSERTS  
LEAKAGE RATES AFTER THERMAL CYCLING-CC/HR.

	Leakage--	After Three		After Induction
	As Molded <u>Δ 15 Psi</u>	<u>Δ 15 Psi</u>	<u>Δ 40 Psi</u>	Heating of Cycled Specimens <u>Δ 40 Psi</u>
Sandblasted and Degreased Sample	0 cc/hr.	0.003	0.043	0.055
Coated with TFE Primer	0 cc/hr.	0.145	0.313	0.030

These data also demonstrate the improvement that induction heating will give to a TFE primed surface by producing a melt fusion with the primer coat. This same improvement was not observed with an unprimed sandblasted and degreased sample.

All of the samples prepared and tested consisted of smooth 1/8 inch diameter inserts. Some improvement in bonding may be realized if a groove is cut in the insert within the area of encapsulation as follows:

Figure 4

Suggested Groove Designs



GROOVES



PROJECTIONS





Several configurations could be used. This groove would provide a means of locking the insert in place so that greater mechanical strength would be exhibited and perhaps better resistance to vibration and pull-through forces would result. This design consideration would be particularly advantageous if the application required a very short length of encapsulation.

#### a. Applications

The technique of sealing metal inserts by direct encapsulation through injection or compression molding appears particularly applicable to a variety of electrical connector and capacitor applications. Terminal pins can be encapsulated and sealed to prevent moisture penetration. The excellent insulating and non-wetting properties of FEP resin are ideal in these electrical applications.

A successful application of this technique consisted of a conductivity cell used to measure the concentration of chemical solutions. Two metal inserts were encapsulated by injection molding of FEP resin. In this application, the inserts were not pretreated nor preheated. As molded, a leak was detected under a 20-psi pressure drop. After post-molding induction heating, however, no leak occurred at 100 psi, either before or after 15 cycles between room temperature and 300° F.

#### B. Sealing to Outer Metal Shell

##### 1. Press Fitting

Press fitting oversize rod stock of FEP resin into a metal sleeve will give a tight interference fit. At least a 5 to 10 mil interference should be maintained after the insert is chilled to about -65° F. At low temperatures shrinkage will occur, and if the sample is press-fitted cold, expansion on warming to room temperature will produce an even tighter fit.

Attempts to achieve a melt bond, after press fitting, by heating the metal sleeves with an induction heater were unsuccessful. Some fusion to the wall was obtained with a surface primed with "Teflon", but never completely around the periphery of the inside diameter so as to give a leak-proof system. The problem was shrinkage of the resin when cooling from the melt state which tended to pull the bond away from the metal wall.



Pressure applied to both ends of the insert during the induction heating process did not help.

A variation of the press fitting technique is to elongate oversize rod stock of FEP approximately 30 to 60% to reduce the diameter. Elongation should preferably be done at elevated temperatures (200 to 300° F.). Stresses imposed by orientation are relieved after the rod stock is press-fitted into the shell and reheated. Relaxation and return to the large diameter results in a stress that gives a tight fit.

A further modification was found beneficial. As shown below, a groove was cut in the sleeve which helped to lock the rod stock within the shell.

Figure 5  
Relaxation of Oriented Rod

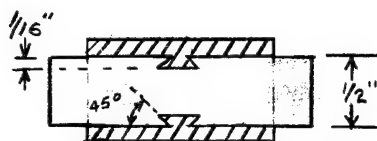


When the oriented rod is relaxed, the resin flows into the groove forming a seal with good mechanical resistance to vibration and pull-through forces. These stresses can be removed, as with the other mechanical seals, if exposed to cycling; air leakage will then result even though the insert is locked within the groove. A post induction heating operation would improve performance; the groove in this case helping to prevent excessive melt flow.

## 2. Molding

A design which incorporates a dovetail projection on the inside wall of the sleeve was found best for achieving good seals to the outer jacket. The dovetail is encapsulated with FEP resin by injection molding. The configuration is shown on the following page.

Figure 6  
Dovetail Design



The interior surface was coated with a "Teflon" primer. The shell was preheated in the mold before filling with resin. In this design, melt shrinkage of resin provided pressure against the surface of the dovetail.

"As molded" samples exhibited varying leakage rates. Incomplete bonding occurred during molding. In all cases, however, induction heating caused fusion to the primed surface and gave a good seal. Samples were water quenched after induction heating to reduce excessive melt flow. Very little change in leakage rate was noted after three thermal cycles. Leakage data for two samples are shown below:

TABLE III

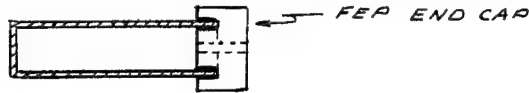
DOVETAIL ENCAPSULATION - LEAKAGE RATES

<u>"As Molded Leakage"</u>	<u>After Induction Heating</u>	<u>After Three Thermal Cycles</u>
4.9 cc/hr. ( $\Delta$ 15 psi)	0.022 cc/hr. ( $\Delta$ 40 psi)	0.025 cc/hr. ( $\Delta$ 40 psi)
6 cc/hr. ( $\Delta$ 15 psi)	0.029 cc/hr. ( $\Delta$ 40 psi)	0.026 cc/hr. ( $\Delta$ 40 psi)

The depth of undercut of a dovetail should be sufficient to permit the melt of FEP resin to flow into and around the projection to effect an encapsulation. Ample surface area is needed if melt fusion is to be achieved with adequate mechanical strength. An understanding of the shrinkage of FEP resin from the melt temperature will help determine satisfactory designs.

Another approach to sealing FEP resin to an outer sleeve is to encapsulate the ends of the sleeve with resin. An example of this technique follows.

Figure 7  
End-Cap Encapsulation



The same principle applies. Shrinkage of resin gives pressure against the metal to effect a bond. If a melt bond does not occur during molding, induction heating can be used as was successfully performed with the dovetail projections.

In each of the techniques described above where FEP resin was successfully bonded to metal shapes, an encapsulation was made. The thermal expansion and contraction of FEP resin compared to metal is very high. It is this difference, therefore, which gives us adequate pressure for bonding to the surfaces of the metal inserts encapsulated by FEP resin. If the thickness of the insert is very large in comparison to the resin thickness, high stresses are present which can result in cracking. Non-uniform resin thicknesses could further aggravate this condition. We experienced no cracking with the designs discussed in this report even after thermal cycling except when a knurled pin was encapsulated and when a pin was induction heated immediately after molding before the FEP resin completely solidified. Incorporating fillers such as glass fiber will greatly improve the stress crack resistance of FEP resin. No evaluations of bonding filled compositions were made. We anticipate that although these compositions may be more difficult to melt bond, satisfactory fusion may be obtainable for many applications.

#### Applications

The techniques described above seem particularly suited to a number of electrical applications where an insulating material with excellent electrical properties and a good seal are required. Capacitors and connectors are typical examples where FEP resin seems best suited. A complete assembly could be made by encapsulating single or multiple pins and simultaneously filling a metal shell with a molding process.

Our discussion of sealing to an outer jacket with FEP resin has been limited to solid inserts. A slightly different approach would be used if tubing of FEP resin were to be sealed to an outer jacket. Heat and pressure are necessary. However, a projection for encapsulating resin in this instance would not be required if air pressure could be inserted into the tube and applied while the resin cooled from the melt temperature.

### III. SPECIAL TECHNIQUES

#### A. Spin Welding

A practical and rapid technique for melt bonding cylindrical mating parts fabricated from FEP resin is by spin welding. The method consists of rotating one part at high speeds against the mating surface to generate frictional heat. When sufficient heat accumulates to develop an interfacial film of molten resin, motion is stopped and the film solidifies to a solid weld.

Despite the high melting point and low coefficient of friction of "Teflon", completed welds on prototype mating cups exhibited tensile strengths roughly 60% as great as the wall sections being joined. Cups joined by this technique are shown below.



Figure 8  
Spin Welded Cups

The required time to spin weld is only a few seconds. The cycle consists of three phases: 1) spin--frictional heat generated melts mating surfaces, 2) transition--halt relative motion of contacting surfaces, 3) cool--molten plastic solidifies under pressure.

The minimum peripheral speed required to generate sufficient heat was found to be 20 to 25 feet per second (3,000 rpm). Even at this speed, however, considerable abrasion and flaking of powdered resin occur during the spin phase before a melt is generated. Application of a thin film of annealing oil to the mating surface helps to minimize flaking and promotes uniform flash of melt. In all probability, higher rotational speeds will also minimize the flaking problem.

In the transition phase, it is important that the relative motion between contacting parts be stopped almost instantaneously. Otherwise, molten film solidifies prematurely and shears. The use of a brake on the powered spindle of the machinery can be helpful in accomplishing this objective.

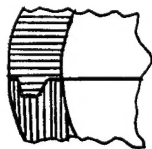
Actual contact pressure on the joint applied during the cooling phase was not measured. Generally, the application of hand pressure proved sufficient.

An important consideration in achieving successful bonds with FEP resin by spin welding is the design of the mating surfaces. End use requirements will influence joint design. As end use becomes severe through pressure, impact or strength, the joints must be strengthened. For lighter duty, the joint design can be relatively simple. The joint design should allow for maximum surface contact area. Since FEP resin is very flexible in thin sections, it is important that the joint be designed for maximum rigidity to assure that mating surfaces will rub continuously and generate sufficient frictional heat. A few typical joint designs are shown below. In experimental work with FEP resin, the tongue and groove joint was used.

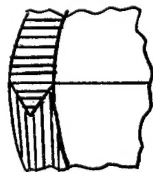
Figure 9  
Spin Weld Joints

Rugged Duty

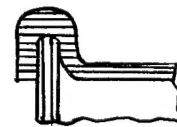
Tongue and  
Groove



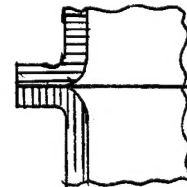
"V"



Overlap



Butt



Light Duty

Spin welding of FEP containers might well lend itself to applications where encapsulation of a liquid or solid is desired, but where the high melt temperature of injection molding cannot be tolerated. With spin welding, the high heat to achieve a melt bond is confined to the mating surface.

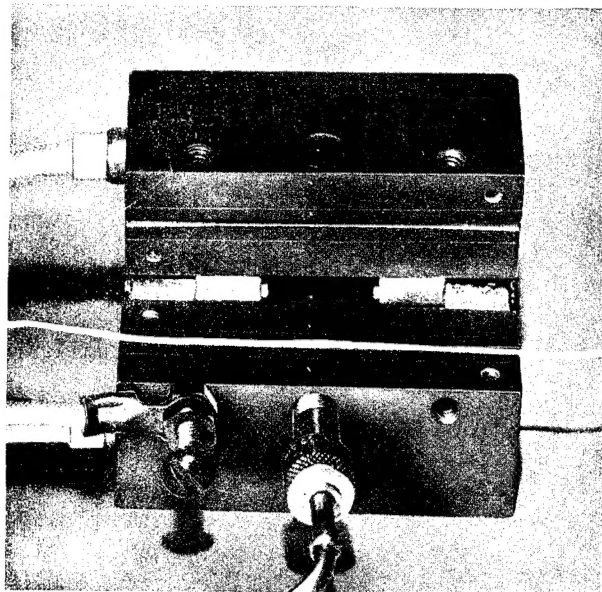
#### B. Film Wrap Process

The availability of FEP film as manufactured by the Film Department of Du Pont provides a simple and useful technique for producing protective coatings. The process involves wrapping the object to be coated while stretching or orienting the film. Upon heating, the film shrinks and conforms to the shape of the object; with continued heating, the tape melts and fuses giving a continuous film. After fusion, it is impossible to see the outlines of the film.

Two-mil FEP film appears to be the most convenient thickness for use in film wrapping. Orientation during the wrapping operation should not exceed 50%, otherwise the film will tend to pull apart during fusion. The film will not adhere to itself during wrapping, and therefore should be held taut. The wrap can be conveniently ended by merely heat sealing the loose end to the underwrap. A 50% overlay should be maintained to assure complete fusion to the underwraps upon heating.

A split-mold die heated by cartridge heaters is a convenient portable means of fusing a film wrap. An example of a mold used in patching wire insulation is shown in Figure 10.

Figure 10  
Split Mold Die



A temperature of 600° F. is desirable. Exposure time to completely fuse the film will vary with the thickness of wrap and size of part being coated. Hand pressure applied to close the mold tightly is generally sufficient to provide uniform heat distribution and complete fusion. The split in the die allows for excessive melt flow. Any flash occurring is easily trimmed. The interior surface of the die should be chrome plated to produce a smooth surface on the finished piece and reduce the probability of sticking in the mold. A silicone mold lubricant or talc applied to the inside mold surface will also help to reduce sticking.

### 1. Applications

The film wrap fusion technique is suitable to a number of applications. For instance, the technique was used successfully in patching wire insulation of FEP resin. Three passes of 2-mil film were made across the area to be patched. The patches were fused at 600° F. in about 10 to 15 seconds. Patches made with this technique were dielectrically sound and scarcely detectable.

Another application using this technique involved encapsulation of a highly sensitive thermocouple probe used in thermal analytical measurements of rate of reactions. Two 36 gage wires projecting from the end of the thermocouple well are brazed together to form the probe. The probe must be protected from the corrosive liquids and still retain high sensitivity in measuring temperature. FEP resin has the ideal combination of properties for this application. The film wrap fusion technique was quite successful.

Encapsulation of electrical components such as diodes is readily performed by this technique. Seals at "U" shaped and "T" shaped branchouts on electrical harness constructions where an FEP resin jacket is used is another application successfully scouted.

### C. Hot Gas Welding

Melt bonding of FEP to itself has been demonstrated with a number of techniques. Hot-gas welding is another means of achieving the melt bond and is most readily adapted to heat sealing of thin sheeting and film of "Teflon" 100. The advantages of this technique include a minimum of equipment, cost and skill. It is readily adapted to field operation and is particularly suited to large operations where heat sealing in a mold is impractical.

Nitrogen may be used as the gas in the welding gun and should be heated to about 900° F. It is important that both contacting surfaces to be bonded be heated to the softening point.



This technique seems particularly suitable to welding seams of sheeting used in tank or other lining applications.

#### D. Heat Shrinking

"Heat shrinkable" tubing of "Teflon" 100, now commercially available, is a special product useful in obtaining mechanical seal encapsulations. "Heat shrinkable" tubing is extruded tubing that has been expanded in diameter through the application of heat and pressure. The tubing is set in its expanded condition but will shrink to its original dimensions upon reheating.

Tubing of "Teflon" 100 can be expanded at any desired temperature below the melt temperature, but 300° F. is frequently used. Reheating to shrink the tubing must be conducted at a temperature equal or greater than the expansion temperature. The thermal properties of the material to be jacketed or encapsulated may dictate the expansion temperature. This technique, therefore, may be more attractive than film wrap fusion process in those applications where the temperatures required to achieve a melt bond cannot be tolerated.

To obtain a good tight mechanical seal, it is important that the pre-expanded diameter of the tubing be smaller than the object to be encapsulated. On shrinking the tubing to its original diameter, a tight fit will result. The amount of shrinkage and, therefore, the tightness of the jacket that occur will depend on the temperature and exposure time used for reheating.

#### 1. Applications

This technique is adaptable to a number of applications. For example, it would be suitable in forming a tight-fitting jacket for wire harnesses, preparing protective coverings for electrical components, providing an antistick coating for heating and cooling coils and in making shrink-on sleeve bearing surfaces. The technique is not primarily one for achieving good seals since it is purely mechanical and at best water tight.

Reminder: The information contained in this report may be claimed in one or more existing patents. We cannot warrant that the use of this information will avoid infringement of all patents. The receipt of this information is not to be construed as a grant of a license under any Du Pont patent. The Du Pont Company assumes no obligation or liability for any advice furnished or for any results obtained with respect to this information.

EAS:cf